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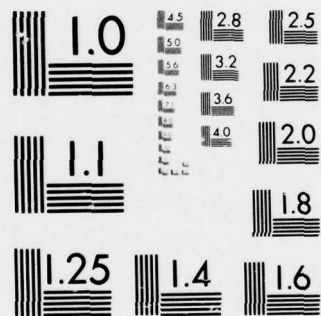
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**MICROPLASTICITY, TENSILE FAILURE,
AND INDENTATION DAMAGE IN
UNFLAWED POLYCRYSTALLINE ALUMINA**

by
James Lankford, Jr.

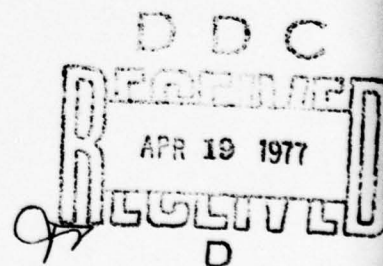
TECHNICAL REPORT

ONR Contract No. N00014-75-C-0668
ONR Contract Authority NR 032-553/1-3-75(471)
SwRI Project No. 02-4231

for
Office of Naval Research
Arlington, Virginia 22217

by
Southwest Research Institute
San Antonio, Texas

April 6, 1977



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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) MICROPLASTICITY, TENSILE FAILURE, AND INDENTATION DAMAGE IN UNFLAWED POLY- CRYSTALLINE ALUMINA		5. TYPE OF REPORT & PERIOD COVERED Interim Report, 1 Apr 76 - 28 Feb 77
7. AUTHOR(s) James Lankford, Jr		6. PERFORMING ORG. REPORT NUMBER SWRI 62-4231-001
9. PERFORMING ORGANIZATION NAME AND ADDRESS Southwest Research Institute P. O. Drawer 28510 San Antonio, Texas 78284		8. CONTRACT OR GRANT NUMBER(s) N00014-75-C-0668
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research 800 North Quincy Arlington, Virginia 22217		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR 032-553/1-3-75(471)
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 23p.		12. REPORT DATE 6 Apr 1977
16. DISTRIBUTION STATEMENT (of this Report) Distribution is unlimited.		13. NUMBER OF PAGES 18 + prelims
		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) DISTRIBUTION STATEMENT A Approved for public release; Distribution Unlimited		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Microplasticity Thermal Activation Tensile Strength Acoustic Emission Indentation Microcrack Initiation Twinning		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Scanning electron microscopy and acoustic emission has been used to study microfracture events occurring prior to tensile failure of unflawed Al_2O_3 tested at 23° and 410°C. A strengthening effect at the latter temperature is shown to be caused by multiple twinning, and enhanced twinning in general. Twinning is found to be responsible for crack initiation at room temperature. In other experiments, SEM was used to observe microfracture events in Al_2O_3 caused by sharp indenters. It was found that lateral crack formation was		

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20. ABSTRACT (Cont'd)

nucleated by mechanical twins located along the inner walls of the indents. The possible relationship of this process to erosion is discussed.

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FOREWORD

The program on which this report is based is oriented primarily toward assessing the role of compressive stresses and compression-induced damage in the failure of brittle ceramics. During the course of extensive compression testing of polycrystalline alumina, it began to appear likely that the microplastic mechanisms responsible for the observed thermally-activated compressive strength behavior might be responsible for certain aspects of tensile and indentation-related fracture as well. This report summarizes the results of experiments designed to explore these ideas further. A second report will be issued within a few months, in which the results of recent compressive failure and other indentation tests will be presented.

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TENSILE FAILURE OF UNFLAWED POLYCRYSTALLINE Al_2O_3

Scanning electron microscopy and acoustic emission are used to investigate the initial stages of tensile failure in unflawed polycrystalline alumina. It is found that deformation twinning plays an important role in crack initiation even at low homologous temperatures, and that the temperature-dependent strength behavior between 23° to 410°C is controlled by twinning.

1. Introduction

Polycrystalline aluminum oxide is generally considered to be an arch-type brittle ceramic. Yet for many years, it has been known that the tensile strength of Al_2O_3 is characterized by certain behavior occurring at relatively low homologous temperatures whose basis has been laid to at least limited plasticity. Generally, this plasticity has been related to thermally-activated events at the tips of subcritical cracks.

The kind of strength behavior under discussion is shown in Fig. 1 [1], in which it can be seen that the bend strength first decreases with increasing temperature until around 400°C, above which point a dramatic increase in strength is observed. It has been suggested [2-4] that the strength increase might be caused by thermally-enhanced local crack tip plasticity, causing crack blunting or bifurcation. However, detailed TEM study by Weiderhorn, Hockey, and Roberts [5] has demonstrated conclusively the absence of dislocations at the tips of arrested tensile cracks in Al_2O_3 . Manifestation of the strength minimum in vacuo, moreover, indicates that the effect is not environmental. It has been proposed [5] that some sort of thermally-activated complexing at crack tips might provide an explanation, but the possible structure and strength-enhancing mechanism of this "complex" has been discussed only sketchily.

Recently the writer reported the results of experiments in which the compressive strength of a polycrystalline aluminum oxide was determined as a function of strain rate and temperature [6]. The presence of a strength minimum at 200-300°C was reminiscent of the similar effect described above for the tensile case. This suggested that the same mechanism (twin-nucleated microcracking) responsible for the compressive strength minimum might explain the tensile strength behavior as well. Indeed, cursory investigation of a few tensile fractures supported this idea. In this paper, the results of a more detailed look at tensile failure in Lucalox are reported, and are shown to support the suggestion that the strength minimum in unflawed Al_2O_3 is caused by thermally-activated deformation twinning, which in turn controls crack initiation.

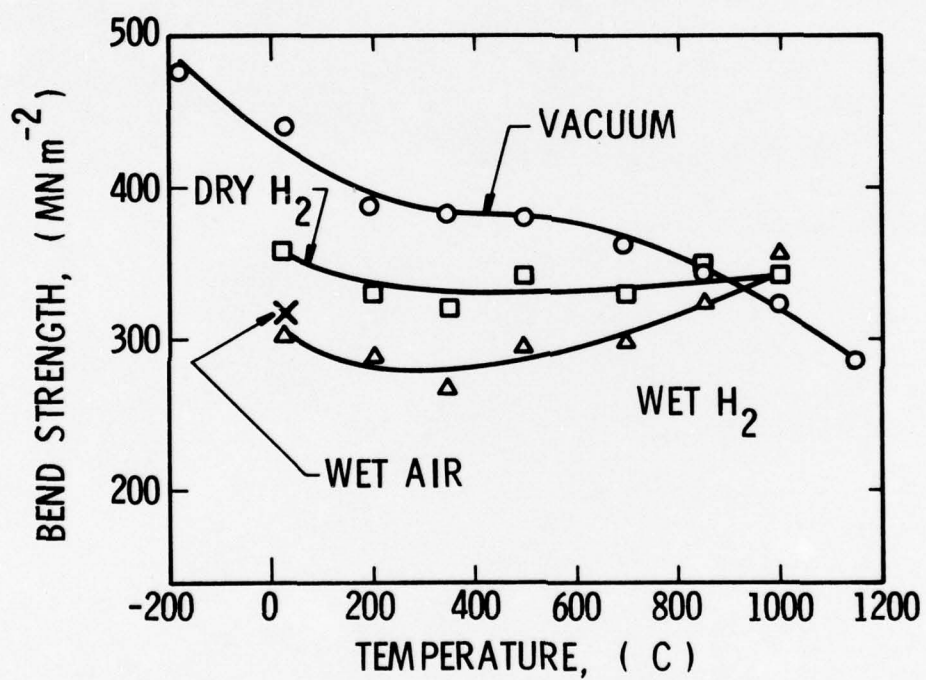


FIGURE 1. TEMPERATURE-ATMOSPHERE EFFECTS ON THE BEND STRENGTH OF LUCALOX [1]

2. Experimental Procedure

As-received rods of polycrystalline alumina (type GW Lucalox*) were cut to length for three-point bending tests, and their ends ground flat for attachment of an acoustic emission transducer. Prior to testing, specimens were cleaned ultrasonically and rinsed in alcohol and distilled water.

Bend tests were carried out at a strain rate of $1 \times 10^{-5} \text{ sec}^{-1}$, at temperatures of 23° and 410°C. Acoustic emission within the frequency domain 100 kHz to 1 MHz was monitored during room temperature testing, using a PZT transducer resonant at 160 kHz (the acoustic emission apparatus and procedures are discussed in detail elsewhere [7]). The acoustic emission detection sensitivity level used in the tensile tests was identical to that used in similar compression tests [7]. The purpose of the three-point bend tests, as opposed to four-point, was to localize any tensile damage and thereby facilitate its detection visually. Similarly, the major goal of the acoustic emission work was to determine the stress level at which tensile damage commenced. In addition, it will be seen that the acoustic emission is helpful in actually assessing the failure micromechanism.

Most of the specimens were tested to failure and then studied in the SEM. A few were loaded to varying sub-failure stress levels and then unloaded for study of early damage. All specimens were coated with palladium for SEM observation.

3. Results

3.1 Acoustic Emission

A particularly instructive format for utilizing the acoustic emission results is to plot the log of the count rate dN/dt versus the log of applied stress σ , as shown in Fig. 2. Here, in addition, are shown earlier tensile results by Evans, Linzer, and Russell [8], for as-machined Lucalox, and recent compressive results [7] for the same as-fired Lucalox used in the present study. In all three cases shown, the strain rates and test temperatures are approximately the same. For the tensile-loaded, as-machined material, the slope S of $\log dN/dt$ versus $\log \sigma$ is essentially constant. On the other hand, $\log dN/dt$ - $\log \sigma$ curve for the as-fired material loaded in tension is divided into two regions of varying slope. The initial value of S is considerably lower, by a factor of 50%, then for the as-machined Lucalox. At higher stresses, a transition occurs, with S approximately equal to the as-machined value. It is interesting to observe that during early stages of damage, S for the as-fired alumina seems to be independent of the mode of loading, i.e., the slope is approximately equal for both compressive and the first stage of tensile loading. However, both the threshold stress (σ_{AE}) for acoustic emission in tension, and also the initial count rate, are approximately an order of magnitude lower than the corresponding parameters in compression.

*General Electric Lamp Glass Division, Cleveland, Ohio.

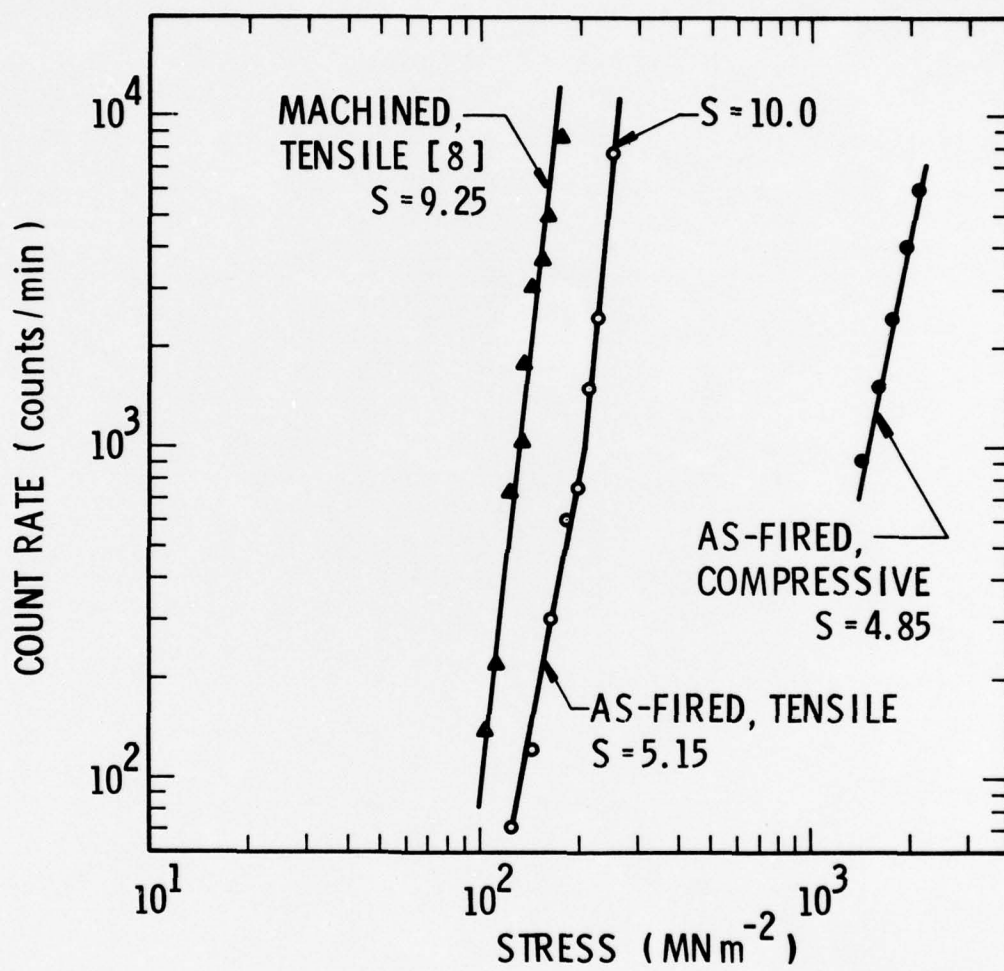


FIGURE 2. ACOUSTIC EMISSION COUNT RATE VERSUS APPLIED STRESS FOR LUCALOX

Bend strength (σ_T) and acoustic emission threshold stress results are summarized in Table 1. The average strength level at 410°C is only slightly lower than that at 23°C, and according to the results of Fig. 1, should reflect the presence of whatever micromechanism is responsible for the general strength increase with rising temperature.

TABLE 1. BEND TEST RESULTS

Temperature (°C)	σ_T (MN m ⁻²)	σ_{AE} (MN m ⁻²)
23	215 ± 23	102 ± 17
410	177 ± 25	---

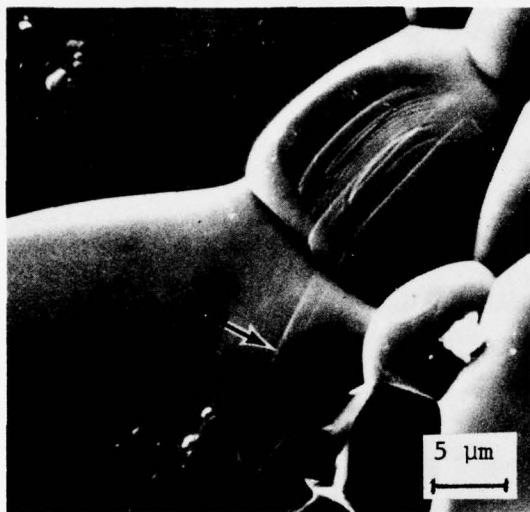
3.2 Scanning Electron Microscopy

Prefracture deformation is more difficult to locate at 23°C than at the higher temperature. However, a diligent scanning of regions adjoining the fracture surfaces of specimens failed at room temperature does reveal evidence of microplasticity and indicates its role in the failure process as well.

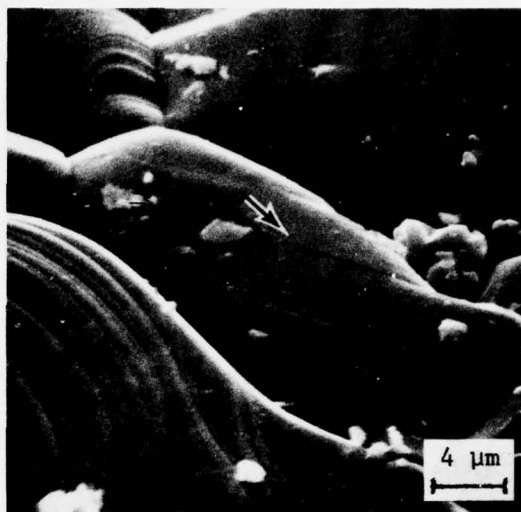
In Fig. 3, several examples of microplasticity produced by uniaxial tensile (bending) stresses at 23°C can be seen. These areas all were located within a few grain diameters of the fracture surface (in each of the photomicrographs to be discussed, the stress axis is vertical). Whether these particular markings reflect twinning or slip cannot be determined with certainty, since they are so narrow. However, they do resemble closely surface deformation produced in the same material by compressive loading, and positively identified as twinning [6]. A comparison between such defects caused by tensile and compressive stresses is shown in Figs. 4a and 4b, respectively. Identification of the compressive flaws as twins was facilitated by the fact that they often are reasonably broad and permit observation of offsets in surface scratches. The tensile twins, particularly at room temperature, seem to be much thinner.

The possible role of the twins in causing tensile failure is suggested by Fig. 5. For the bend specimen shown in Fig. 5a, the apparent fracture origin was the thumbnail region indicated by the arrow. This region was immediately opposite the center support in the three-point bend rig. Near the center of the thumbnail, the grain shown in Fig. 5b was located. This grain exhibits the same twin-like features noted previously, with the fracture plane appearing to wander along several twin planes. Such behavior has been observed during compressive microfracture of Lucalox, and is typical of twin-nucleated cracking [6] in this material.

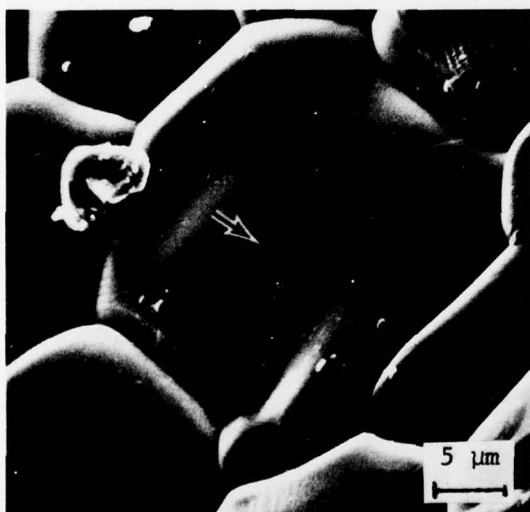
Similar twinning and twin-related cracking is found for tensile tests carried out at higher temperatures. In Figs. 6 and 7, extensive



(a)



(b)

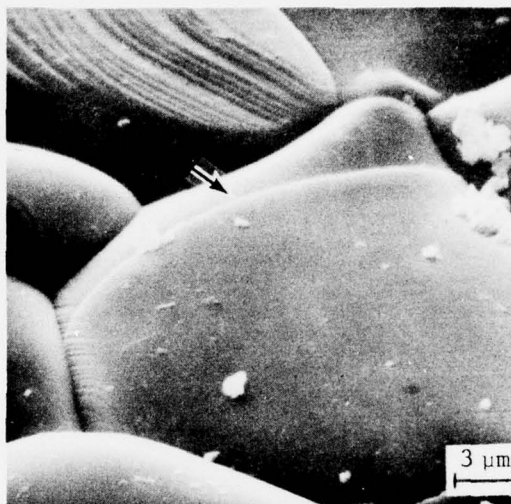


(c)

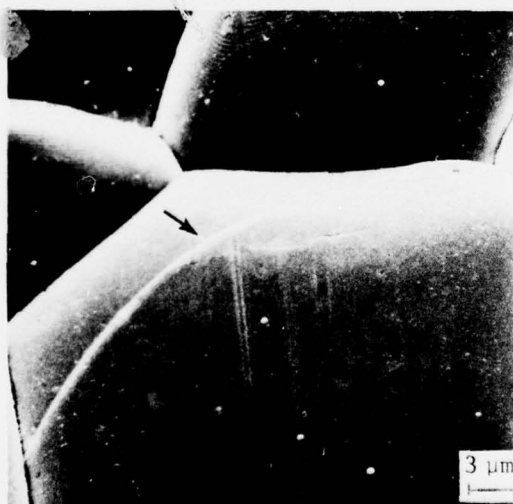


(d)

FIGURE 3. MICROPLASTICITY (ARROWS) PRODUCED NEAR TENSILE FRACTURE SURFACE AT 23°C

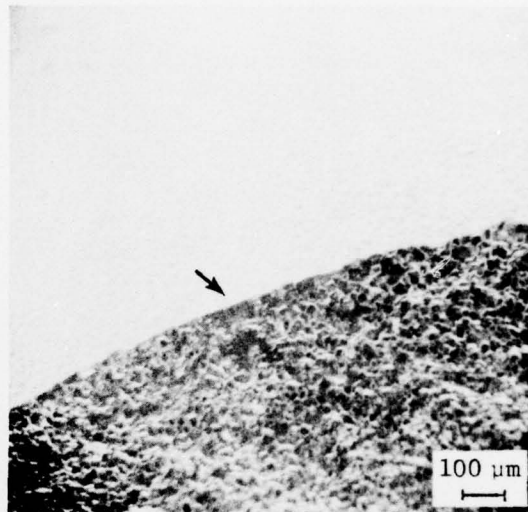


(a) Tensile

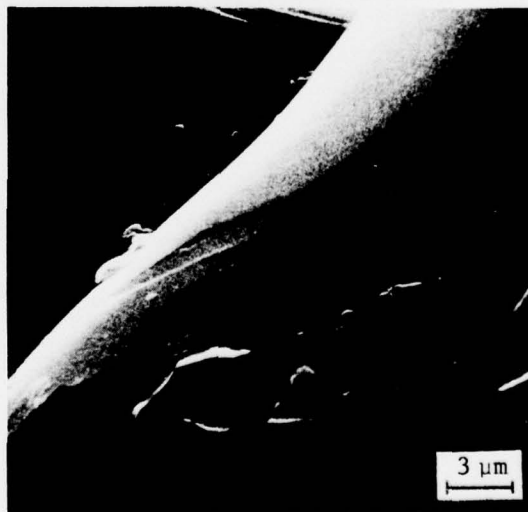


(b) Compressive

FIGURE 4. COMPARISON OF SURFACE MICROPLASTICITY (ARROWS)
PRODUCED IN TENSION AND COMPRESSION AT 23°C



(a) Fracture surface; arrow indicates origin of failure



(b) Grain associated with initial flaw, showing apparent twin-induced microfracture

FIGURE 5. RELATIONSHIP BETWEEN TWINNING, MICROFRACTURE, AND TENSILE FAILURE

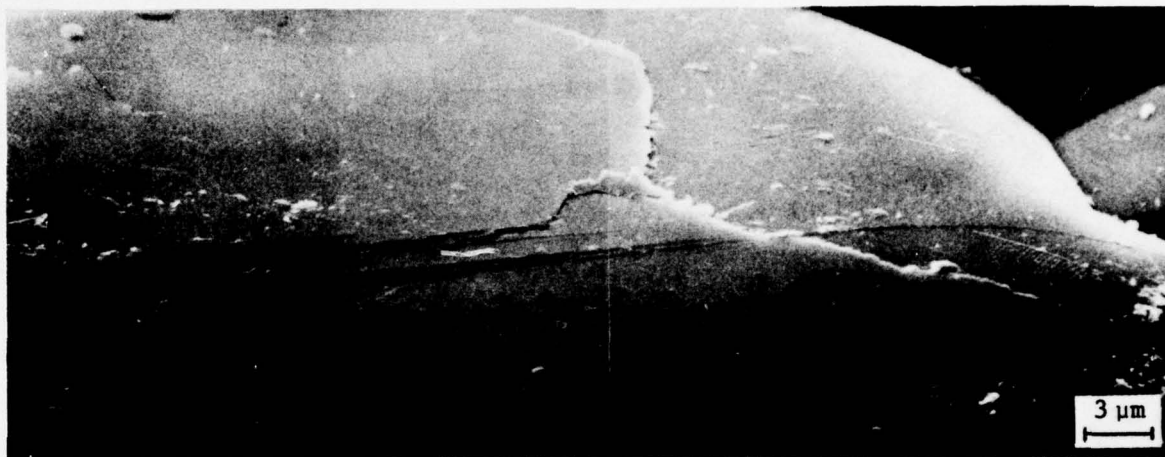


FIGURE 6. TWINNING UNDER TENSILE LOADING AT 410°C

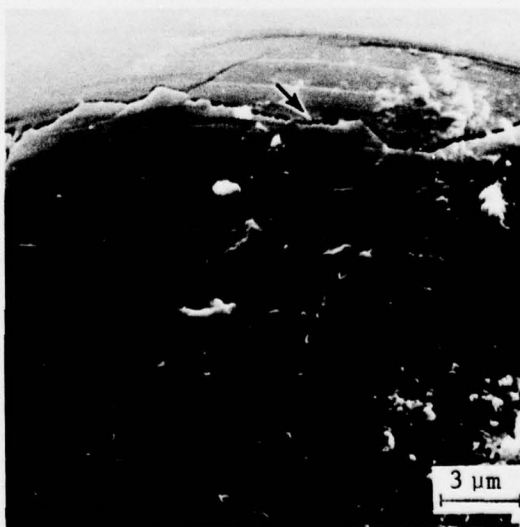


FIGURE 7. TWINNING AND TWIN-PLANE MICROCRACKING (ARROW) UNDER TENSION AT 410°C

fine-scale twinning has occurred in grains adjacent to the major fracture surface, with microcracks having initiated within some of the twins (note the wandering of the crack (C) in Fig. 6b from one twin plane to another). Also at higher temperature, it is observed that multiple twin systems begin to be activated, as was the case during compression testing [7]. This is shown in Fig. 8, where intersecting twins (T) have initiated cracks within (C1) and normal to (C2) twin traces. Some of the twins nucleated at 410°C achieve a significantly greater thickness than those formed at room temperature. These twin facets were often noted near failure nucleation sites; such a location is shown in Fig. 9, in which the local fracture has wandered along a possible twin facet (arrow).

4. Discussion

The first indication that some mechanism other than microcracking alone is responsible for the observed thermally-activated tensile strength behavior can be deduced from the acoustic emission results. Earlier, Evans, et al. [8], had developed an analytical model for acoustic emission due solely to microcracking in bulk specimens. Based on this model, the slope of $\ln(dN/dt)$ versus $\ln\sigma$ should be approximately 10. This was found to be the case for their as-machined Lucalox specimens, which contained a large population of prior flaws, i.e., machining cracks, and which gave a slope of 11.7 (Fig. 1). In the present case, the much lower initial slope and the markedly reduced count rate for equivalent stresses point to the operation of some mechanism other than microcracking to account for the early stages of acoustic emission in unflawed Al_2O_3 . As noted by Evans, et al. [8], the only other obvious candidates are dislocation motion and twinning. The fact that the compressive and early stages of tensile stressing of unflawed Lucalox produce essentially the same $\ln(dN/dt)$ versus $\ln\sigma$ slope implies that the source of acoustic emission is identical in both cases. SEM work previously has proven twinning to be the earliest source during compression [6], and the present electron optical observations are compatible with the same conclusions for tensile failure. However, it is more difficult to separate twinning from possible slip traces in the latter case, since the apparent tensile twins are not as thick as those generated during compression.

Certain work by others is in general agreement with the idea that twinning controls tensile fracture in cases such as the present one. More than ten years ago, Heuer [9] showed that deformation twinning was associated with fracture initiation in corundum single crystals broken in bending at temperatures as low as -196°C. Later, Congleton, et al. [2] showed that there exists a minimum at 250°C in the tensile fracture stress of polycrystalline specimens containing drilled holes. Two possible explanations for this minimum were offered: (1) temperature-dependent crack tip plasticity, or (2) thermally activated crack initiation involving slip or twinning. The TEM work by Weiderhorn, et al. [5], discussed earlier argues against the former. Recently, Becher [10] has shown that the tensile failure of as-ground sapphire bars is controlled by grinding-induced deformation twins, along the habit planes of which the initial microfractures propagate. The implication here is that while grinding introduces several forms of surface damage, including microcracking, slip, and twinning, the propensity for the twin/parent interface to fail under tensile loading, combined with the

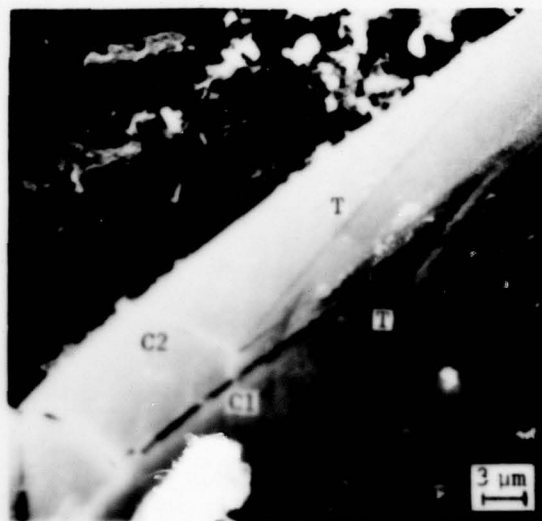


FIGURE 8. MULTIPLE TWIN SYSTEMS ACTIVATED IN TENSION AT 410°C. Intersecting twins produce cracks within (C1) and normal to (C2) twin traces.

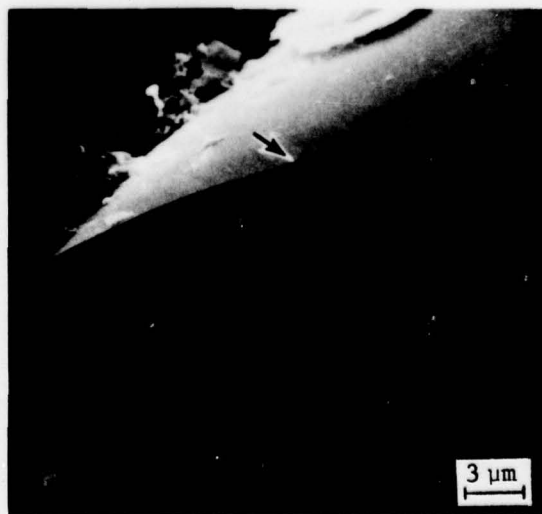


FIGURE 9. POSSIBLE TWIN FACET (ARROW) NEAR TENSILE FRACTURE ORIGIN (T = 410°C)

size of the twins, can cause twinning to dominate as the principal failure mechanism. Finally, deformation markings similar to some of those seen in the present study have been observed by Coble and Parikh [11] on Lucalox deformed in tension. However, the markings at that time could not be unambiguously associated with failure, and their identification as twins was only tentative. The present results indicate the correctness of these earlier speculations [11].

It is interesting to consider the stress levels apparently required for twin nucleation in tension and in compression. Acoustic emission indicates that twinning during tensile loading ($T = 23^{\circ}\text{C}$, $\dot{\epsilon} \sim 10^{-5} \text{ sec}^{-1}$) commences at around 130 MN m^{-2} , while compressive loading under similar conditions requires a stress of 1400 MN m^{-2} to initiate twins, as shown in Fig. 2. The reason for this ten-fold strength differential may be related to the inherent stability of flaws nucleated under compression as compared to those formed in tension.

It is possible that during compressive deformation, microtwins actually nucleate at stress levels significantly lower than those detected by the present acoustic emission system. However, it is already well known [6] that flaws such as microcracks, once nucleated in compression, are exceptionally stable and frequently require much higher stress excursions in order to extend. If the stress wave displacement due to initial compressive twinning were below the minimum displacement sensitivity of the acoustic emission system, as might be the case for very small microtwins, then the presence of the twins would be undetected. Detectable acoustic emission would then occur at higher stresses when the twins are driven completely across grains, which is generally accomplished without cracking the twin/parent boundary; rather, it is more common in compression [6] for cracks to nucleate at twin/grain boundary intersections. Usually the twins are stable enough to grow sidewise to achieve a finite thickness. On the other hand, microtwins formed in tension may be less stable. They usually are very thin, tend to extend completely across grains, and often are microcracked along the twin/parent boundary. A suggested scenario is one in which the first twins nucleated in tension zip across a grain as soon as they are formed, accompanied by stress wave emission. Upon reaching a critical size on the order of the grain size, the weak twin/parent interface frequently parts in tension, which effectively precludes further thickening of the twin. At higher stress levels, these cracked twins themselves begin to serve as active flaws, causing crack initiation and extension in adjacent grains. Thus, they can from this level on be considered as "microcracks," whose acoustic emission response undergoes a change from that characteristic of twinning to that corresponding to microcrack extension. This is manifested in the S value for higher stresses being approximately equal to that found by Evans, et al. [8], for initially microcracked (machined) specimens.

The fact that twinning begins to occur on multiple systems at higher temperatures explains the observed temperature dependent strengthening effect. Plastic accommodation at local stress concentrations through multiple twinning reduces the necessity for crack formation. Moreover, cracks which do occur in such multiple twin fields tend to interfere and interact with one another rather than running neatly across each twinned grain. This

could reduce the propensity for further crack extension in such regions, thereby effectively raising the failure stress.

5. Conclusions

The temperature-dependent strength behavior of unflawed polycrystalline alumina in the temperature range 23°C to 410°C is caused by deformation twinning through its role in microcrack initiation. The twinning is responsible for acoustic emission, whose count rate is dependent upon stress according to the same relationship observed for twinning in compression. The strengthening effect at higher temperatures is caused by enhanced twinning generally, and multiple twin system activity in particular, which aids in accommodating local strain concentration.

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TWINNING AND MICROCRACK NUCLEATION DURING INDENTATION OF ALUMINUM OXIDE

1. Introduction

Indentation fracture recently has been characterized in terms of the sequence of formation and the morphologies of cracks produced during point indentation of brittle solids [1,2]. TEM observations by Hockey [3] and Hockey and Lawn [4] have demonstrated the presence of micro-plastic damage zones beneath indentations in several brittle materials, and Hurley [5] has observed slip-like surface features adjacent to indentations. Hockey [3] has obtained surface terraces, on etching indent sites, whose directions correspond to possible slip and twin traces. However, the relationship between plastic deformation and crack initiation remains unclear, and presently only the extension of certain types of indentation cracks can be predicted, using the fracture mechanics approach [6].

During penetration by a sharp indenter, two kinds of cracks are nucleated; the first to form, known as radial or median vent cracks, extend into the solid along indent diagonals. Initiation occurs on the loading half-cycle, within the inelastic subsurface zone. On the unloading half-cycle, other cracks are nucleated near the surface, in the vicinity of the elastic-plastic boundary. Growth of these lateral cracks is away from the indent and roughly parallel to the surface before curving up to intersect it. Of these two types of fracture, lateral cracking is thought to be relevant to material removal during solid particle erosion.

It has been suggested by Evans and Wilshaw [6] that lateral cracking in particular may be initiated by flaws generated during plastic deformation of the region subsurface to the indenter (this suggestion is supported by the independence of the lateral crack formation load with respect to surface condition). It is the purpose of this report to demonstrate the validity of this idea for alumina.

2. Experiments

As-fired Lucalox rods were indented statically with a diamond pyramid indenter. Individual grains, of average dimension 40 μm , were selected for multiple indentation at sequentially higher loads, with the indent orientation maintained constant within a given grain. Following indentation, the specimens were coated with palladium for study in the scanning electron microscope.

3. Results and Discussion

For loads of 10 gm and less, indentations are formed with no observable (at 20,000X) cracking. However, faint evidence of plasticity is visible along the inner walls of the indentation, in the form of a regular pattern of banding. These bands are more readily visible at higher loads, as shown

in Fig. 1, where other features also are of interest. Radial cracking begins at loads in excess of 10 gm, with the radial cracks being associated with the indentation corners; they do not appear to extend to the bottom of the indent until loads of several hundred gm are attained.

Plastic "deformation bands" are found along the indentation walls, as well as on the specimen surface adjacent to the indent, as shown in Fig. 1 (arrows). Recent work [7] indicates that twinning is the earliest stage of damage in this same material when stressed in uniaxial compression or tension at 23°C. It also is known that indentation produces a compressive stress state on the indent facets, and tension in the surrounding surface region [6]. Hence, it is likely that the observed indentation plasticity consists of deformation twins. In support of this suggestion, Hockey [8] has observed through TEM that whereas the bulk of the plastic damage in sapphire is confined below the indentation, microtwins often extend upward from this region to the surface.

It has been shown [7] that the twin planes in Lucalox are preferred fracture planes, along which cracks may initiate in both tension and compression. Under indentation loading, they appear to serve as lateral crack nuclei for loads of 100 gm and greater. In Fig. 2, a lateral crack has formed at the left edge of the indent crater, with the center section of the crack, along the edge of the indent, being exceptionally straight. Rotation of the viewing angle by 90° (Fig. 3) shows that this section corresponds to the intersection of an apparent deformation twin with the rim of the indent, below which other non-cracked twins (probably lenticular in shape [3]) are visible. The crack extends laterally over a distance of about 5 μm , as determined by viewing the specimen optically and focusing below the surface to image the crack plane. The conceptual relationship between twinning and crack formation is sketched in Fig. 4.

This type of deformation-induced cracking was observed consistently, its repeatability being further evidence of the role played by the indentation plastic zone, as opposed to pre-existing flaws. For a given crystallite and indenter orientation, the crack initiation pattern at multiple indents was almost identical, i.e., the initial lateral cracking always occurred on a specific, preferred side of each indent, and each crack usually could be identified with a twin trace. At higher loads, lateral cracks begin to nucleate at other sides, also in a repeatable sequence.

The fact that the twins required for lateral crack initiation are present early on implies that the critical load for macrocrack formation does not correspond to that needed to nucleate a twin. Rather, it is more likely that the occurrence of a critical microcrack extension load is based on the statistical probability of crack extension along the twin plane, which increases with the volume of stressed material [6]. The critical extension load is a function of grain orientation relative to the indenter, since this factor controls the angular relationship between the unloading tensile stress field and the most favorable twin plane, on which the twins have formed during the loading indent cycle. This is reflected in variation in the lateral crack extension load by approximately 50% for varying indenter/crystallite orientations.

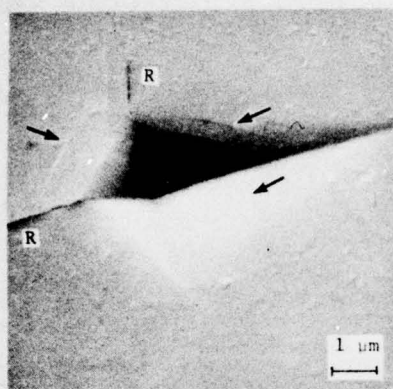


FIG. 1

Indentation due to 50 gm load, with radial cracks (R) and twins (arrows).

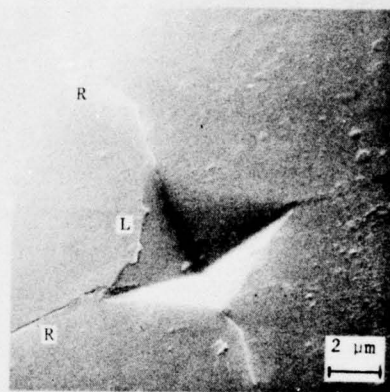


FIG. 2

Indentation due to 100 gm load, with radial (R) and lateral (L) cracks.

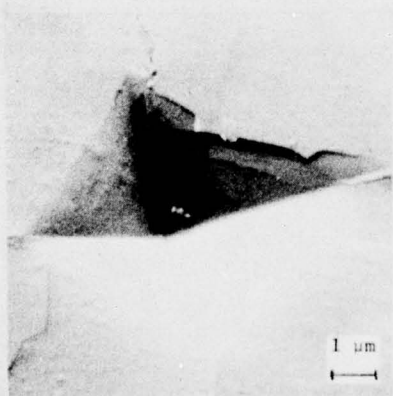


FIG. 3

Magnified, rotated view of Fig. 2, showing relationship between lateral crack and twins (curved slightly due to inelastic indent relaxation).

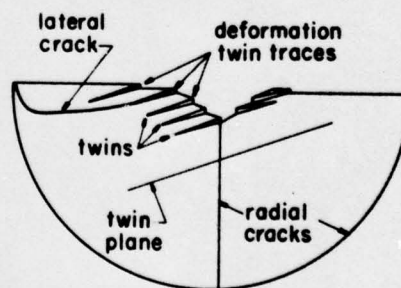


FIG. 4

Conceptual relationship between indentation twins and lateral crack formation.

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